Application Note AN-RS-008

Identification of monomers with Raman spectroscopy

Monitor the polymerization process from monomer to polymer

Polymers are comprised of macromolecules that are in turn made of numerous identical or similar structural units, referred to as monomers. The number of monomers, including additives or inhibitors such as benzoquinone – used to endow the polymers with specific properties – is enormous.
All polymer manufacturers use the same monomers and can profit from a quick raw material check before feeding them into the polymerization process. Raman spectroscopy provides a nondestructive, in-situ, real-time analytical method to monitor the polymerization process by tracking monomer consumption and polymer formation. Ultimately, Raman spectroscopy is a convenient and efficient tool for various industries related to the polymer sector.

INTRODUCTION

This Application Note demonstrates the convenient identification of monomers within seconds using Raman spectroscopy. Monomers such as styrene, various alkyl methacrylates, vinyl acetate, ethylene glycol, phenol, terephthalic acid, and urea, as well as additives or inhibitors like benzoquinone, can be identified rapidly and unambiguously.

A quick demonstration of the distinct spectra of different monomers and their respective polymer leads to an in-depth look at the polymerization reaction of Bakelite.

EXPERIMENTAL

Raman spectroscopy is a simple point-and-shoot nondestructive analysis technique that allows for fast and safe analysis with no sample preparation. In some cases, samples can even be analyzed in their original packaging.

A handheld 785 nm Raman device, featuring an automated workflow and Orbital Raster Scanning (ORSTM), was used to collect basic monomer spectra. The polymerization reaction of Bakelite was safely monitored by placing a laboratory Raman fiber-optic probe against the wall of a beaker containing the reactants, allowing real-time evaluation as the reaction progressed.

MONOMER SPECTRA

Figure 1 contains overlaid spectra of different monomers (and benzoquinone), demonstrating that Raman is both sensitive and high specific—it is very easy to distinguish the spectra of different materials.

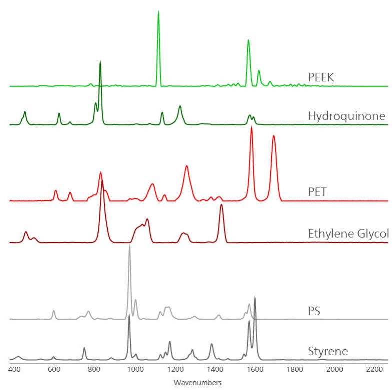

Figure 1. Overlay of Raman spectra from the analyzed monomers and benzoquinone.

MONOMERS AND POLYMERS

Monomers bind together to form polymers during the polymerization process. Real-time monitoring of the polymerization reaction with Raman spectroscopy is a powerful way to optimize and control the process and the resulting product. It is Raman's specificity that permits monomers to be easily distinguished from their respective polymers. Figure 2 illustrates the spectral differences between polymer starting materials and products.

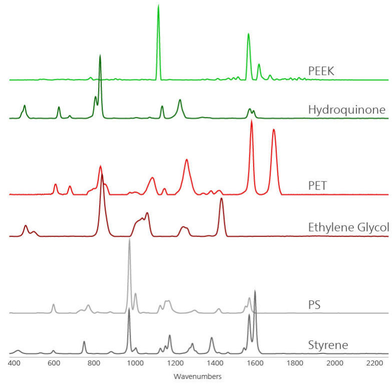

Figure 2. Overlay of Raman spectra from monomers with their respective polymers, including polyether ether ketone (PEEK), polyethylene terephthalate (PET), and polystyrene (PS).

POLYMERIZATION MONITORING WITH RAMAN

Bakelite is a thermosetting plastic created through the polymerization of phenol and formaldehyde. Figure 3 shows how the Raman peaks of phenol diminish as it reacts with formaldehyde to form a cross-linked polymer, while new peaks emerge due to changes in the vibrational environment. High Raman shift regions permit the observation of changes in the C-H stretching vibrations of phenol (2000–4500 cm–1), providing insight into this reaction.

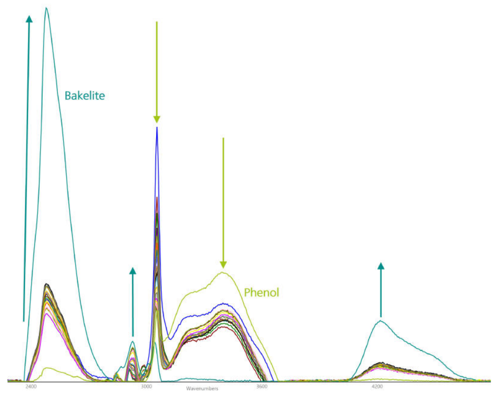

Figure 3. Evolution of Raman bands for phenol and Bakelite during the polymerization reaction.

CONCLUSION

Real-time, on-site analysis using Raman spectroscopy enables polymer manufacturers to maintain product integrity through quality checks at every stage, from raw materials to final products. This ensures consistent quality, optimizes efficiency, and drives innovation in polymer manufacturing.

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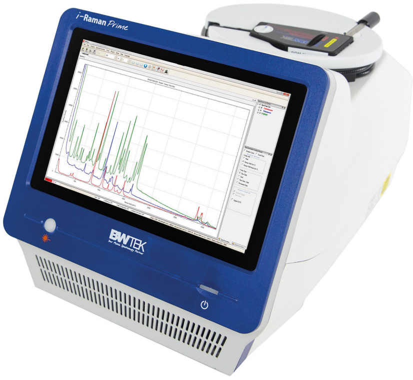
CONFIGURATION

MIRA P Advanced

The Metrohm Instant Raman Analyzer (MIRA) P is a high-performance, handheld Raman spectrometer used for rapid, nondestructive determination and verification of different material types, such as Pharmaceutical APIs and excipients. Despite the small size of the instrument, the MIRA P has a ruggedized design and features a high-efficiency spectrograph design equipped with our unique Orbital-Raster-Scan (ORS) technology. The MIRA P is fully compliant with FDA 21 CFR Part 11 regulations.

The Advanced Package includes an attachment lens for analyzing materials directly or through containers (laser class 3b), as well as a vial holder attachment for analyzing samples contained in glass vials (laser class 1).

CONFIGURATION



i-Raman Prime 785H Portable Raman Spectrometer

The i-Raman® Prime 785H is a low-noise, high-throughput, and fully integrated Raman system with an embedded tablet computer and a fiber-optic sampling probe. Using a high-quantum-efficiency CCD array detector with TE deep cooling (-25 °C) and high dynamic range, this portable Raman spectrometer delivers research-grade Raman analysis capabilities, including real-time quantitation and identification. The high throughput gives Raman spectra with excellent signal-to-noise ratio, making it possible to measure rapid processes and to measure even the slightest Raman signals, detecting subtle sample differences.

In addition to its portable construction, the i-Raman Prime 785H features the unique combination of wide spectral coverage and high resolution, thus enabling measurements from 150 cm^{-1} to 2,800 cm^{-1}. The i-Raman Prime can be battery-operated for easy portability, providing research-grade Raman analysis capabilities for high-precision qualitative and quantitative work wherever needed. The system is optimized for use with our STRaman® technology for analyses through non-transparent packaging.